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Mineralogical and Microscopic Evaluation of Coarse Taconite Tailings from Minnesota Taconite Operations

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Abstract

Eighteen coarse taconite tailings samples were collected in 2000-2001 from five western Mesabi Range taconite (iron ore) operations located in northern Minnesota, i.e., EVTAC, Hibbing Taconite (Hibtac), USX Minntac, Ispat Inland (Minorca), and National Steel Pellet Company (NSPC), to test their physical, geological, chemical, and mineralogical properties (Zanko et al., 2003). The goal was to assemble a body of technical data that could be used to better assess the potential of using a crushed taconite mining byproduct like coarse tailings for more widespread construction aggregate purposes, primarily in roads and highways. An important part of the mineralogical assessment included X-ray diffraction (XRD) analyses and microscopic evaluation of the size and shape (morphological) characteristics of potentially respirable microscopic mineral particles and fragments.

Quantitative mineralogy, based on XRD analyses, showed that the dominant mineral in all samples was quartz (55 to 60 percent), followed by much smaller amounts of iron oxides, carbonates, and silicates. Specialized microscopic analyses and testing performed by the RJ Lee Group, Monroeville, PA, on both pulverized (-200 mesh, or 0.075 mm) and as-is sample composites showed that no regulated asbestos minerals or amphibole minerals were present. A very small number of mineral cleavage fragments/mineral fibers were detected, but these were mostly minnesotaite, a silicate mineral common to the Biwabik Iron Formation. Amphibole minerals, absent in coarse tailings samples from the five western Mesabi Range taconite operations, were present in a single eastern Biwabik Iron Formation sample collected in 2003 for Lake County from the Cliffs Northshore operation in Silver Bay, MN.

Importantly, the *Superfund Method for the Determination of Releasable Asbestos in Soils and Bulk Materials*, EPA 540-R-97-028 (1997), as modified by Berman and Kolk (2000) failed to generate any protocol fibers, i.e., fibers longer than 5µm and thinner than 0.5µm, from either the western coarse tailings samples or the single eastern Biwabik Iron Formation sample. The combined findings suggest coarse tailings and other taconite mining byproducts should be treated with the same common sense safety and industrial hygiene approach practiced for all mineral-based materials that have the potential to generate respirable dust.

Keywords: Taconite; Amphibole; Biwabik Iron Formation; Mesabi Iron Range

Note: Changes of ownership have occurred at two of the taconite operations. First, National Steel was purchased by U.S. Steel, and National Steel Pellet Company (NSPC) has been re-named; it is now called Keewatin Taconite. Second, EVTAC Mining Co. was purchased by Cleveland-Cliffs, Inc. and China's Laiwu Steel Group, and is now called United Taconite.

1. Aggregate potential of taconite mining byproducts

Over 50 million short tons of aggregate are used annually in Minnesota, with 75% coming from sand and gravel sources, and the remaining 25% from crushed stone sources. Recent studies by Southwick et al. (2000) and the Aggregate Resources Task Force (1998; 2000), have addressed the growing pressure on aggregate resources in the State of Minnesota, particularly in the seven-county Twin Cities (Minneapolis and Saint Paul) Metro Area. There, aggregate shortages have been predicted within the next 10 to 15 years, as currently permitted reserves near exhaustion, and as residential and commercial developments expand and encroach on remaining aggregate resources. Other areas of the state, like the Highway 61 corridor along the north shore of Lake Superior, have also had difficulty supplying new sources of high quality crushed aggregate for local construction jobs. Southeastern Minnesota carbonate (limestone and dolomite) quarries have experienced similar pressures for providing quality construction aggregates that meet Superpave (SUPERior PERforming asphalt PAVement) specifications. Compounding these resource pressures, the permitting of pit and quarry expansions and new aggregate mines has become more complex and difficult, environmentally *and* socially, given the growing “not in my back yard” (NIMBY) reaction to such projects.

Millions of tons of taconite mining byproducts are generated annually by Minnesota's taconite mining operations, and coarse tailings are one of the largest components. Because these byproducts represent a potentially huge alternative to conventional crushed stone aggregates, a project was funded by the Minnesota Local Road Research Board (LRRB) to conduct a more detailed evaluation of coarse taconite tailings (Zanko et al., 2003). Despite being used extensively for road and other construction applications at the mines, coarse tailings usage beyond the confines of the mining properties has generally been limited to local projects. This is unfortunate, because byproducts like coarse tailings are the end result of tremendous expenditures of energy and capital used in mining and processing taconite. If one or more taconite mining byproduct like coarse tailings are found to be suitable for uses in large-scale road and construction aggregate applications, and are demonstrably superior to other types of aggregate, then the economic and environmental benefit to the taconite companies and the State of Minnesota could be significant, given their potential to offset some of the impending aggregate shortages just discussed.

The project's main goal, therefore, was to generate and document, following a one-year sampling program, the technical data needed for making such determinations, i.e., relating geological, mineralogical, and chemical properties to potential aggregate performance, and to address some of the important economic issues like transportation. It was also believed that the lack of well-documented technical information on several fronts, such as the potential health risk posed by inhalation of taconite dust, may have been contributing to perceptions about taconite

mining byproducts like coarse tailings that were needlessly limiting their use potential. With respect to this latter point, a more detailed mineralogical evaluation of the samples was conducted during the project. The evaluation consisted of qualitative and quantitative mineralogy, plus specialized microscopic analyses and testing. The findings are summarized and discussed in this paper.

2. Taconite mine locations

Figure 1 shows the relative location of each taconite operation on Minnesota's Mesabi Range. The Biwabik Iron Formation (BIF), which runs diagonally from southwest to northeast on the map, is the source of ore for the mines. Coarse tailings generated at the five westernmost operations, i.e., Ispat Inland (Minorca), EVTAC (now United Taconite), Minntac, Hibbing Taconite (Hibtac), and National Steel Pellet Company (NSPC; now Keewatin Taconite), were the focus of the study. The two easternmost taconite facilities, i.e., Northshore Mining Company in Babbitt and Silver Bay, and the now closed Cliffs Erie (LTV Steel) operation in Hoyt Lakes, were not part of the study.

3. Taconite tailings

Davis (1964) describes taconite as a hard, dense rock, composed largely of an intimate mixture of silicates and very fine magnetite (Fe_3O_4) crystals. Because magnetite constitutes less than one-third of the weight of crude taconite ore, large amounts of tailings are generated during the taconite process. Taconite tailings range in size from clay (less than 2 microns) to coarse sand ($3/8$ in., or about 10 mm), and are produced during various stages of crude taconite ore beneficiation. They are highly siliceous and contain a low percentage (3-5 percent) of iron in the magnetic form, i.e., magnetite.

Tailings are typically described as being fine or coarse. *Fine tailings* are composed of extremely fine rock particles, more than 90 percent of which are smaller than 0.003 in. (0.075 mm, or -200 mesh); they have little practical use at the mines, and end up in tailings basins. *Coarse tailings*, conversely, contain less than 10 percent fines, i.e., particles smaller than 0.003 in. (0.075 mm, or -200 mesh), and their top size is about $3/8$ in. (10 mm, or -4 mesh). Coarse tailings are used for mine-site projects like tailings basin containment cells, dikes, dams, as construction fill material, and for building and maintaining haul roads and shovel pads.

Coarse tailings generally meet the construction industry's definition of *fine aggregate*, i.e., aggregate particles less than $3/8$ in. As a byproduct of crushing and grinding, they are angular (100 percent fractured faces) and comparable to manufactured fine aggregates used widely in construction (N. Whiting, pers. comm., 2002). Consequently, the physical properties that make coarse taconite tailings suitable for heavy-duty applications at the mines suggest expanded use in local, state, and federal highway construction projects. That is why coarse tailings from taconite mining operations were the focus of the research project.

EVTAC, Ispat Inland, and Minntac have separate coarse and fine tailings circuits in their plants, whereas no separation of coarse and fine tailings occurs at the Hibtac and NSPC plants. At EVTAC, Ispat Inland, and Minntac, coarse tailings are produced from cobber tailings (non-magnetics) and are separated from the fine fraction with spiral classifiers.

Coarse tailings are *not* separated from fine tailings in the Hibtac and NSPC concentrators. Combined fine and coarse tailings flow as slurry to the tailings basin in two flumes at Hibtac,

and through a pipe at NSPC. However, some size separation is achieved as the larger particles settle out of the slurries near the points of entry to the basins. Over time, the tailings build up, much like sediments in a river delta, and periodically this buildup of coarser tailings is excavated and placed in piles. At Hibtac, a dragline removes and stockpiles these coarse tailings when they tend to dam the entry to the basin; at NSPC, a bulldozer pushes the built-up of coarse tailings out of the channel that leads into the basin.

4. Sample collection

Coarse tailing samples from the five western taconite operations were acquired on a quarterly basis over a one-year period, beginning in the Fall of 2000. Following their acquisition, samples were prepared by the NRRI's Coleraine Minerals Research Laboratory (CMRL) for all subsequent project testing. Tailings from EVTAC, Ispat Inland, and Hibtac were sampled by CMRL research technicians; at Minntac and NSPC, the samples were collected by plant personnel. Sufficient sample was collected at each operation to fill one plastic-lined 55-gallon drum. Each drum contained about 450 lbs. (200 kg) of sample.

Samples were typically collected during an 8-hour shift; at Minntac, samples were collected over several days and shifts. The goal was to collect samples that were reasonably representative not only of the tailings, but of portions of the iron-formation from which the ore originated, i.e., multiple shovel positions located throughout each mine. Admittedly, characterizing coarse tailings from all five taconite operations was a compromise in sampling practicality, given that about 30 *million* long tons of coarse tailings would be generated during the sample collection year, which translates into over 80,000 long tons *per day*. However, it is believed that the project's sampling strategy not only provided materials that reflected the typical range of taconite ore blends processed by each company during a production year, it provided materials that would actually be used by potential aggregate consumers. Table 1 shows the sample collection dates, and the corresponding sample numbers used throughout the project.

The eighteen samples were collected at the following mine locations:

- EVTAC: at the conveyor discharge above the truck pocket.
- Hibtac: from the tailings basin at recently deposited dragline stockpiles.
- Minntac: directly from spiral classifier discharge points.
- Ispat Inland (Minorca): at a conveyor discharge point inside the concentrator building.
- NSPC: from piles excavated from of the channel that leads into the tailings basin.

5. Sample preparation

At CMRL each sample was dumped into a rotating hopper that fed an eight-way rotary splitter. The eight splits were dumped back into the drum twice to blend each sample before representative portions were removed for testing and analyses.

After another pass through the rotary splitter, the one-eighth portions were adjusted using a Jones riffle. This step provided two 50-pound samples for testing by Mn/DOT, one 50-pound and six 25-pound samples for aggregate testing by Braun Intertec Engineering, Inc., and a 25-pound head sample for processing at CMRL. All sample pails were tagged with sample

identification and all drums and pails were tightly closed with lids to retain the moisture that was present when sampled at each taconite facility. Mn/DOT samples were delivered to the District 1 facility in Duluth. Braun Intertec, having a facility relatively nearby in Hibbing, picked up their samples at CMRL.

CMRL head samples were weighed, dried at 110°C, and weighed to determine moisture percentages. After the dried samples were mixed, approximately 1,000 grams were split out (riffled) for size analyses, and approximately 2,000 grams were split out for chemical analyses and X-ray diffraction (XRD). The latter were further prepared by standard CMRL procedures, i.e., they were stage crushed in a roll crusher to pass 20 mesh, and 150 gram to 200 gram portions were split out and stage pulverized in a Braun pulverizer to pass a 200 mesh screen. About 20 grams were split and bagged for XRD, and the balance of pulverized samples were submitted for chemical analyses. Samples were similarly prepared and composited for specialized microscopy.

6. Size analyses

CMRL conducted particle size (sieve) analyses on each coarse tails sample. The data, composited for each mine, are presented in Figure 2. The analyses show that a minimal amount of fines (-200 mesh material) is present in most of the coarse tailings samples. This is especially true for EVTAC, Minntac, and Ispat Inland, where coarse and fine tailings are separated. In fact, their size distribution plots are very similar. Overall, samples from Hibtac and NSPC contain a larger percentage of particles finer than 0.5 mm, while EVTAC and Minntac samples contain a smaller percentage.

Note how similarly the gradations plot for EVTAC, Minntac, and Ispat Inland (Minorca), and the minimal amount (about 2.5 percent) of -200 mesh material that is present in their samples. The Hibtac and NSPC plots reflect the more variable composition of their coarse tails, and the higher percentage of -200 mesh material. The difference between the size distribution of samples collected at Hibtac and NSPC and those collected from the other three operations (at which coarse and fine tailings are separated) simply reflects the basic differences in each company's process flowsheet. The plots also show that EVTAC and Minntac coarse tailings contain a smaller percentage of particles finer than 1 mm than do coarse tailings from the other companies.

7. Mineralogy

Taconite ore from the Biwabik Iron Formation is composed primarily of quartz and magnetite, plus varying amounts of iron oxides, carbonates, and silicates. To determine the mineralogy of the coarse taconite tailings samples, X-ray diffraction and geochemistry were used.

X-ray diffraction (XRD) is an analytical method used for identifying specific minerals in a rock or soil sample. Minerals will refract x-rays in diagnostic ways, depending on their crystal/molecular structure. The technique is qualitative, not quantitative, but it identifies the major, minor, and trace minerals present in a sample. To determine quantitative mineralogy, XRD must be combined with whole rock chemistry.

X-ray diffraction patterns for the first nine tailing samples were generated by NRRI Economic Geology Group (EGG) personnel on the Philips XRD machine in the University of

Minnesota, Duluth (UMD), Department of Geosciences. The last nine samples were run at the University of Minnesota Characterization Facility at the Shepherd Laboratories in Minneapolis. Figures 3a and 3b are examples of the XRD patterns produced at UMD and Shepherd Laboratories, respectively. The peaks in the patterns showed the presence of various combinations of minerals. Again, this is the *qualitative* aspect of the technique. However, when the patterns were used with reference to major oxide chemical analyses performed by CMRL (Table 2), and total oxides were normalized to 100%, the percentage of each mineral in the sample could be calculated. This is the *quantitative* aspect of the technique. Total iron, or TFe_2O_3 as reported in Table 2, is based on the CMRL Fe% assay, multiplied by 1.42972. Percent sulfur (S%) is also reported, but it is not included in the major oxide total. The sulfur assay is used for determining the sulfide mineral content of the tailings. Finally, the H_2O and CO_2 values, when combined, give the equivalent of loss on ignition (LOI).

A spreadsheet routine developed by one of the authors was used to calculate mineral percentages, based on the XRD patterns and chemical analyses. As each mineral was calculated, its chemical constituents were subtracted from the original analysis or from the residual percentage, so that: 1) the latter was as near zero as possible; and 2) the total of the mineral percentages was at least within 0.5 percent of the total chemical percent, i.e., total oxides. Minerals were calculated as follows:

- Magnetite was determined by the Satmagan magnetic iron measurement.
- Stilpnomelane was calculated assuming all K_2O was present in this mineral.
- Apatite was assumed to be the only P_2O_5 -bearing mineral.
- Ankerite and/or other Ca-bearing carbonates used all CaO.
- Siderite (and/or Rhodochrosite) used the remaining CO_2 and MnO.
- Minnesotaite, Talc, and Greenalite used the remaining MgO and FeO.
- Chamosite was calculated if Al_2O_3 residual was greater than 0.05 percent.
- Hematite (and Goethite) used remaining Fe_2O_3 .
- Quartz was equal to the remaining SiO_2 .
- Rutile equivalent equaled TiO_2 percent.
- Pyrite was calculated using all S.
- Carbon was total C minus carbon as CO_2 .

The chemical compositions of the various minerals (Table 3) were determined from the following sources: Deer et al. (1962); Blake (1965); Klein (1974); and Floran and Papike (1975; 1978). The composite quantitative mineralogy for all 18 samples is illustrated in Figure 4, showing that quartz is by far the dominant mineral, followed by lesser amounts of iron oxides, carbonates, and silicates.

8. Specialized microscopy

Specialized microscopic work was subcontracted to the RJ Lee Group of Monroeville, PA. This work was done, in part, to address concerns and questions about the potential for coarse tailings to generate "asbestiform" particles. These concerns and questions have direct lineage to the Reserve Mining Co. trial of the 1970s, and to the mineralogy of the Biwabik Iron Formation at its eastern end. Bartlett (1980) provides useful detail and background on the issues surrounding the Reserve Mining case

The iron-formation's eastern end was metamorphosed 1.1 billion years ago during the emplacement of a series of mafic igneous intrusions, collectively known as the Duluth Complex, and amphibole minerals were formed. The amphiboles are principally of the cummingtonite-grunerite series, and include some actinolite and hornblende (Ross et al., 1993; French, 1968). These minerals first appear in the iron-formation midway through the former LTV Steel (now Cliffs Erie) property near Hoyt Lakes, as depicted by the diagonal lines in Figure 5. Some of their cleavage fragments can take on a morphology and an aspect ratio (length to width) of >3:1 (and greater than 5 microns in length) that has been called "asbestiform". The presence of amphibole minerals having these morphological characteristics was at the heart of the Reserve Mining Co. trial.

All of the coarse tailings samples studied by Zanko et al. (2003) came from taconite operations well to the west of the amphibole mineral area. Nevertheless, it was believed important to subject the samples to state-of-the-art analytical techniques and methods for detecting asbestos minerals and mineral cleavage fragments because: 1) associations between "taconite tailings" and "asbestos" were still being made, regardless of where in the iron-formation the taconite ore and tailings originated; and 2) the potential use of coarse tailings and other taconite mining byproducts on a more widespread and significant basis inside and outside Minnesota would only generate more questions about those perceived associations. Therefore, the specialized microscopy work was performed to provide a factual and science-based evaluation and documentation of the mineralogical and physical nature of potentially respirable microscopic coarse tailings particle.

9. Specialized microscopy summary for coarse tailings samples from five western Biwabik Iron Formation taconite operations

A combination of X-ray diffraction, polarized light microscopy (PLM), scanning electron microscopy (SEM), and transmission electron microscopy (TEM) was used by the RJ Lee Group to determine the mineralogy and size and shape characteristics of the mineral particles in the coarse tailings samples, and to determine if any asbestos minerals were present. Two sample types were submitted from each taconite operation:

- 1) composited, as-is, samples; and
- 2) composited samples pulverized to pass a 200 mesh screen (-200 mesh).

These composited samples were believed to be reasonably representative of the coarse tailings generated over a single production year at each mine. Excerpts and summaries from the RJ Lee Group's report of analytical methods and testing results follow.

9.1. X-ray powder diffraction

A portion of each pulverized sample was ground in a mortar and pestle, mixed with fluorite (used as an internal standard), and backloaded into a standard XRD holder for analysis. The samples were processed using standard run parameters on a Philips XRD unit equipped with graphite monochromatized copper radiation.

As established in the previous XRD analyses performed by NRRI and the University of Minnesota, the RJ Lee Group's XRD analyses determined that the primary component of all

samples was quartz, with varying amounts of hematite, magnetite, and siderite. The primary layer silicate mineral identified by XRD was minnesotaite. (XRD cannot differentiate between fibrous and cleavage fragment varieties of minerals). No regulated amphibole was observed during these analyses.

9.2. Polarized light microscopy

Both the pulverized samples and the < 200 mesh fraction of the tailings were analyzed for asbestos content using polarized light microscopy (PLM) following the analytical procedures outlined in EPA/600/R-93/116, *Method for the Determination of Asbestos in Bulk Building Materials* (Perkins and Harvey, 1993). Any non-asbestos materials, including cleavage fragments, were also identified and quantified during the PLM analysis. Quantitation of the sample was performed using a 1,000-point count procedure.

Trace levels of non-amphibole cleavage fragments were observed in the Ispat Inland (Minorca) and Minntac samples by PLM. The cleavage fragments (four fragments, total, in the *entire* PLM analyses of all samples) had moderate aspect ratios (greater than 3:1, length/width), but showed no evidence of fibril structure. Based on the PLM analyses, no regulated asbestos minerals were detected.

9.3. Scanning electron microscopy

The pulverized samples and the < 200 mesh fraction of the as-received samples were analyzed using scanning electron microscopy (SEM) in general accordance with the methods outlined in ISO/DIS 14966 (2002). A portion of each sample was weighed and placed in a beaker containing acetone. The suspension was shaken and an aliquot removed and deposited on a polycarbonate filter. A portion of the filter was placed on a carbon planchette and carbon coated (to reduce electrical charging on the particles).

The samples were analyzed in a SEM at an accelerating voltage of 20 KeV at a working distance of 15 – 17 mm. During the analyses, back-scattered electron images and energy dispersive spectroscopy (EDS) were used to evaluate the particles. Typical images and EDS spectra were acquired for major particle types, e.g., quartz, minnesotaite, talc, iron oxides, and carbonates. Back-scattered electron images and EDS spectra were also obtained for particles that had a greater than 3:1 aspect ratio and were greater than 0.25 μm in diameter. Figures 6a-f are examples of images and spectra produced for the samples.

No asbestiform minerals were observed during the SEM analyses. Several cleavage fragments were observed in the < 200 mesh fraction that was sieved from the “as-received” Minorca tailings; no cleavage fragments were observed in the pulverized Minorca sample. The chemistries for the cleavage fragments observed in the Minorca sample are consistent with minnesotaite and talc.

9.4. Transmission electron microscopy/EPA Superfund method

The tailings samples that were pulverized to < 200 mesh were analyzed by transmission electron microscopy (TEM) to determine the weight percent of asbestos and cleavage fragments. The analytical procedure used for the TEM analysis was in general accordance with the analytical portion of ASTM D 5756 (2002). A portion of each sample was weighed and placed

in a beaker, which was then filled to 100 ml with distilled water. The suspension was sonicated for 3 minutes, then allowed to settle for 2 minutes before an aliquot of the supernatant was removed and redeposited onto a 25 mm mixed cellulose ester (MCE) filter. The filter was then prepared for analysis using direct preparation procedures.

No asbestiform minerals or amphibole cleavage fragments were observed during the TEM weight percent analysis.

9.5. EPA Superfund method

The “as-received” samples were analyzed using the *Superfund Method for the Determination of Releasable Asbestos in Soils and Bulk Materials*, EPA 540-R-97-028 (1997), as modified by Berman and Kolk (2000). The samples were sieved through a 1 mm screen (#18) and the two size fractions weighed. Seventy grams of the < 1 mm fraction were placed in the holder and tumbled as described in the method. Several filters of the elutriated sample were collected over varying times until one filter contained approximately 100 µg of samples. This filter from each sample was evaluated in the TEM for protocol fibers (fibers longer than 5 µm and thinner than 0.5 µm).

No asbestiform fibers or cleavage mineral fragments were observed during the TEM analyses of the elutriated samples.

9.6. Significance of specialized microscopy results

Based on the combined results of the various tests, the RJ Lee Group concluded that no asbestos minerals were present in the coarse tailings samples collected from the five western Mesabi Range taconite facilities. The PLM and SEM analyses showed that some mineral cleavage fragments with an aspect ratio of >3:1 were present, but none belonged to the amphibole mineral group. Most of the cleavage fragments were consistent with minnesotaite or talc. As previous mineralogical work has shown, e.g., French (1968), minnesotaite and talc are silicate minerals typically found on the western Mesabi Range. Interestingly, one of the cleavage fragments was silica-rich (quartz/chert?) (Fig. 6e), and another was calcium-rich (calcite?) (Fig. 6f). These results suggest that mineral particles classified as “asbestiform” can be produced by crushing and grinding rocks that contain neither amphibole minerals nor asbestos.

10. Specialized microscopy results for an eastern Biwabik Iron Formation sample

As a point of comparison, Lake County, Minnesota, submitted an eastern Biwabik Iron Formation sample to the RJ Lee Group. The same specialized microscopic analyses and testing methods performed on the western Mesabi Range coarse tailings samples, i.e., PLM, SEM, TEM, and EPA Superfund Method, were done on the eastern sample. Refer to the previous section for specific analytical method descriptions.

The sample, submitted as-is and pulverized to -200 mesh, was a composite of dry cobber lean ore collected in January, 2003, from several locations within the Milepost 7 basin at the Cliffs Northshore taconite operation in Silver Bay, Minnesota. Dry cobber lean ore is a byproduct of the ore processed from Northshore’s taconite mine near Babbitt, Minnesota (refer to Figs. 1 and 5). This byproduct is much coarser (more than 50% of the particles are coarser

than 3/8 in.) than the coarse tailings produced at the five western taconite plants, yet it contains a low percentage of fines (less than 3% finer than 200 mesh). PLM, SEM, and TEM were performed on the sample split pulverized to –200 mesh; the EPA Superfund Method was performed on the as-is sample split.

10.1. Polarized light microscopy

PLM analysis of the pulverized –200 mesh sample showed it contained approximately 10 to 15 percent cleavage amphibole fragments identified as “tremolite/actinolite”. The cleavage fragments were reported to have moderate aspect ratios (>3:1, length/width), but showed no evidence of fibril structure. The presence of amphiboles is consistent with the mineralogy of the metamorphosed eastern end of the Biwabik Iron Formation. The relatively large number of cleavage fragments detected by PLM in the pulverized Northshore sample contrasts with the trace amounts found in samples from the five western operations. The difference suggests that amphibole-bearing taconite from the eastern Biwabik Iron Formation generates cleavage fragments more readily than does the silicate-bearing taconite from the western BIF.

10.2. Scanning electron microscopy

No asbestos minerals were observed in the pulverized –200 mesh sample during the SEM analyses. Cleavage fragments were observed that were consistent with tremolite/actinolite. Figures 7a-d are SEM images and spectra produced for some of the Northshore amphibole cleavage fragments. Note the calcium peaks that are indicative of tremolite and actinolite. Figures 7a and 7b depict blocky cleavage fragments; Figures 7c and 7d depict narrower cleavage fragments.

10.3. Transmission electron microscopy

TEM was used to determine the weight percent of asbestos and cleavage fragments in the pulverized –200 mesh sample. No asbestos minerals were observed during the TEM weight percent analysis. Tremolite/actinolite cleavage fragments were observed at the 0.02 weight percent concentration. According to the RJ Lee Group, the TEM cleavage fragment concentration is said to differ from the PLM concentration due to the size of the cleavage fragments. Because the TEM suspension was allowed to settle for two minutes, particles larger than about 20 µm settle out, and are not analyzed in the TEM. Therefore, the low (0.02 weight percent) indicates that cleavage fragments are primarily larger than this dimension.

10.4. EPA Superfund method

The “as-is” Northshore sample was analyzed using the *Superfund Method for the Determination of Releasable Asbestos in Soils and Bulk Materials*, EPA 540-R-97-028 (1997), as modified by Berman and Kolk (2000). The procedure failed to generate any protocol fibers (fibers longer than 5 µm and thinner than 0.5 µm). No asbestiform minerals or cleavage mineral fragments were observed during the TEM analyses of the elutriated sample.

10.5. Significance of specialized microscopy results for an eastern Biwabik Iron Formation sample

Based on the combined results of the various tests, the RJ Lee Group concluded that no asbestos minerals were present in the sample collected from the Cliffs Northshore property. Amphibole cleavage fragments were detected by both PLM and SEM in the sample split pulverized to -200 mesh. The TEM analyses, in general accordance with the analytical portion of ASTM D 5756 (2002), indicated that nearly all of the cleavage fragments were larger than 20 μm . Lastly, the EPA Superfund method failed to generate protocol fibers from the “as-is” sample split, i.e., the size and type of material that would most likely be used as construction aggregate.

These results appear to show that amphibole-bearing eastern Biwabik Iron Formation taconite mining byproducts, when pulverized to -200 mesh, will generate a larger number of cleavage fragments than comparably pulverized silicate-bearing western Biwabik Iron Formation taconite mining byproducts. In his Ph. D. thesis, Stevenson (1983) determined that the number of amphibole “fibers” generated by the processing of copper-nickel ore from the adjacent Duluth Complex was proportional to the amount of amphibole present in the ore and to the fineness of grind during processing. However, it is important to remember that typical dry cobber lean ore production generates less than 3 percent -200 mesh-sized particles in the final product. This latter point, when viewed with the EPA Superfund Method test results on the “as-is” sample split, indicates a low potential for problematic dust generation if the material is used as construction aggregate. But because only one sample was subjected to the microscopy testing, supplemental testing of additional eastern Biwabik Iron Formation taconite mining byproducts would probably be desirable.

11. MSHA sampling data and Minnesota Department of Health mesothelioma findings

Mineral “fibers” can be present in a variety of geological settings, as revealed in an analysis of air sampling conducted by the Mine Safety and Health Administration (MSHA). Following news reports in 1999 that described serious health problems associated with amphibole asbestos-contaminated vermiculite mined at Libby, Montana (Schneider, 1999), MSHA collected air samples from several mines and quarries in the United States between January 10, 2000, and March 25, 2002. The air sample data summarized herein can be found at MSHA’s web site:

www.msha.gov/asbestos/asbestos.htm.

MSHA’s intent was to assess the air quality at mining and quarrying operations where respirable mineral fibers could be generated, but especially at other U.S. vermiculite mines.

The MSHA tests make no distinction as to mineral type, only to the total number of particles that are $>5 \mu\text{m}$ in length and have a minimum aspect ratio (length:width) of 3:1 as determined by Phase Contrast Microscopy (PCM), in accordance to the National Institute for Occupational Safety and Health (NIOSH), Method 7400, Issue 2 (NIOSH, 1994). The current MSHA personal exposure limit (PEL) is 2.0 fibers/cc for an 8 hour, shift weighted average. The OSHA PEL is much more stringent at 0.1 fibers/cc for an 8 hour, shift weighted average. However, fibers $< 0.25 \mu\text{m}$ in diameter cannot be detected by the PCM method.

Figure 8 summarizes the MSHA air sampling findings by mine/quarry type for which at least six samples were available for averaging. These data are for fiber counts as determined by PCM only. The 0.1 fibers/cc OSHA PEL standard is represented on the graph by the horizontal dashed line. The average PEL is graphed for samples that exceed the detection limit at each mine/quarry type. The number of samples used for determining the average PEL for each mine/quarry type is also reported. The data are taken in their entirety; no distinction is made between the various job categories for which air samples were collected. The data (and graph) show Minnesota iron mines having an average PEL of 0.026 fibers/cc for an 8 hour, shift weighted average. This value is well below OSHA's 0.1 fibers/cc standard, is comparable to the granite average, and is lower than the limestone average. For comparative purposes, samples taken in the 1970s at the Libby, Montana, vermiculite mine measured fiber levels at 18 fibers/cc.

Iron ore mines in Minnesota and Michigan were also sampled more thoroughly, in response not only to the Libby situation, but to two Minnesota Department of Health (MDH) Minnesota Cancer Surveillance System (MCSS) reports published in 1997 (MCSS Epidemiology Report 97-1) and 1999 (MCSS Epidemiology Report 99-2). The MDH reports showed a higher rate (70% higher than expected) of the asbestos-related cancer, mesothelioma, occurring in males in a seven-county region of northeastern Minnesota.

Because some people ascribed the higher cancer rate to taconite dust exposure, the MDH conducted a follow-up study (MCSS Epidemiology Report 03:1, published in 2003) to look specifically at the incidence of mesothelioma in iron miners and possible sources of commercial asbestos exposure in the mining industry. In a recently released summary of that study, Brunner et al. (2003) stated the following:

“Seventeen individuals (all men) diagnosed with mesothelioma in Minnesota between 1988 and 1996 were found to have worked in the iron mining industry. Of the 15 for whom adequate work histories were available, 14 had identifiable sources of exposure to commercial asbestos in jobs they held both inside and outside of the mining industry.”

These findings suggest that exposure to commercial asbestos products may have been a significant factor in the development of mesothelioma in the miners.

13. Conclusions

Mineralogical and specialized microscopic analyses show that coarse taconite tailings sample composites from five western Mesabi Range taconite mines did not contain any of the six regulated asbestos minerals, nor did they contain amphibole minerals. A very small number of cleavage fragments/mineral fibers were detected by SEM, primarily minnesotaite. Amphibole minerals, absent in coarse tailings samples from the five western Mesabi Range taconite operations, were present in the single eastern Biwabik Iron Formation sample submitted by Lake County.

Mineral cleavage fragments can also be generated from many rock types, not just taconite, as demonstrated by the Mine Safety and Health Administration's (MSHA) 2000 to 2002 air sampling data. Importantly, the *Superfund Method for the Determination of Releasable Asbestos in Soils and Bulk Materials*, EPA 540-R-97-028 (1997), as modified by Berman and Kolk (2000) and performed by the RJ Lee Group, failed to generate any protocol fibers from either the western coarse tailings samples or the eastern Biwabik Iron Formation sample.

The specialized microscopy analyses, coupled with the MSHA data and recent Minnesota Department of Health findings, should provide useful information to regulators and potential end-users about the nature of the dust that would be encountered when handling and using taconite mining byproducts like coarse tailings for construction aggregate purposes. The combined results show how geology, mineralogy, chemistry, physical properties, particle size, shape, and morphology, and intended end-uses must all be taken into account when working with any potential dust-generating aggregate, and should be viewed with proper context and perspective.

With respect to coarse taconite tailings, context and perspective includes the following points:

- most of the potentially respirable dust generated during the production of coarse tailings occurs at the mine sites;
- coarse tailings are a byproduct of typically wet mineral processing techniques, and are often transported as a slurry; they are, to varying degrees, a “washed” product;
- coarse tailings contain a small percentage of fines (-200 mesh), especially at operations that make a fine and coarse separation, where the percentage of fines is typically less than 2.5%;
- coarse tailings are hard and resist abrasion;
- coarse tailings are composed primarily of quartz; and
- coarse tailings would most likely be used in buried applications, such as granular fill, or encapsulated in bituminous asphalt or concrete mixes, thereby reducing the potential for post-construction dust exposure to negligible levels.

Therefore, coarse tailings and other taconite mining byproducts should be treated with the same common sense safety and industrial hygiene approach practiced for all mineral-based materials that have the potential to generate respirable dust, silica or otherwise.

Acknowledgments

The Minnesota Local Road Research Board (LRRB), University of Minnesota Center for Transportation Studies (CTS), the Minnesota Department of Transportation (Mn/DOT), and the Natural Resources Research Institute (NRRI) are gratefully acknowledged for providing project funding, support, and guidance. Lake County, Minnesota, is also acknowledged for supporting the analysis of an eastern Mesabi Range taconite byproduct sample. Lastly, Messrs. Keith Rickabaugh and Drew R. Van Orden of the RJ Lee Group, Monroeville, PA, are thanked for their explanations and descriptions of the analytical procedures and testing methods used for evaluating the coarse tailings samples, as reported in this paper.

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Figures and Tables

Figure 1: Taconite mine location map and coarse tailings sources in boxed area (from Zanko et al., 2003).

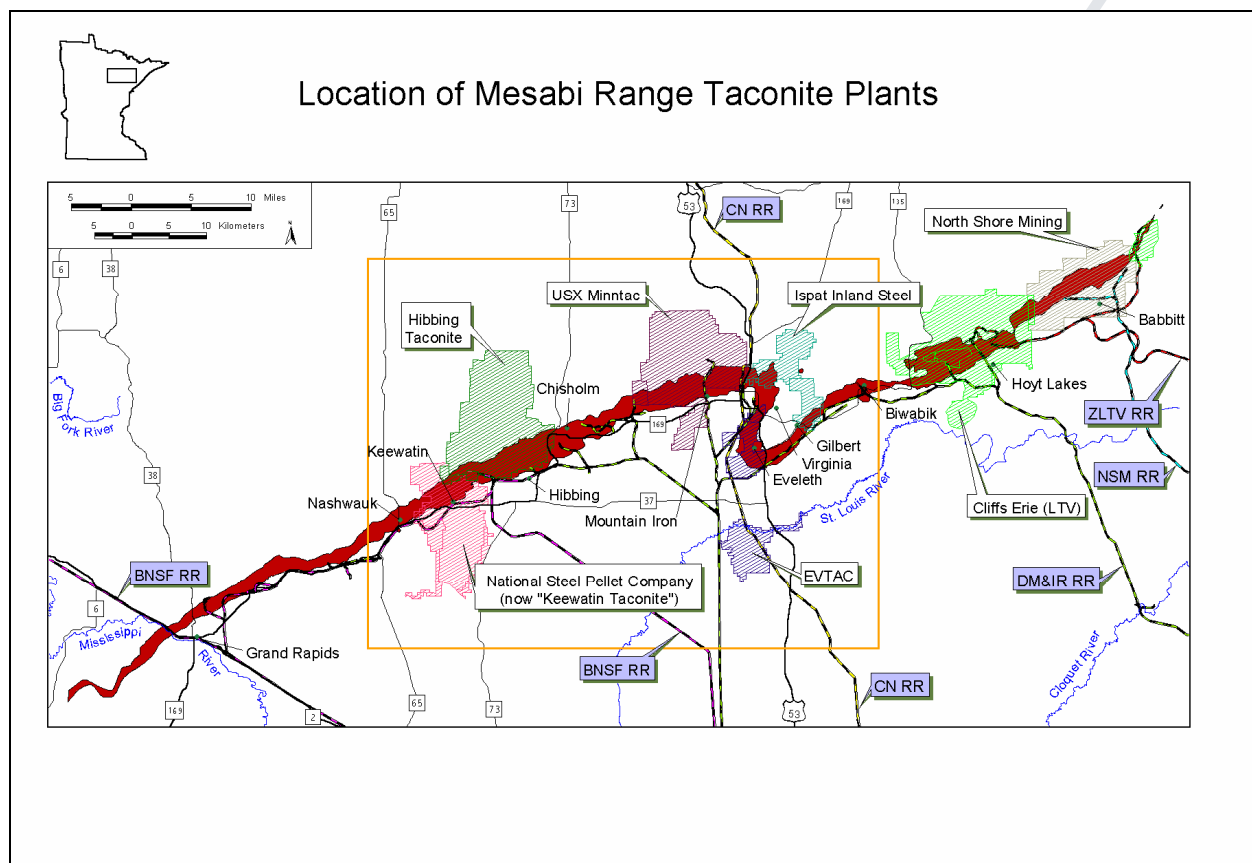


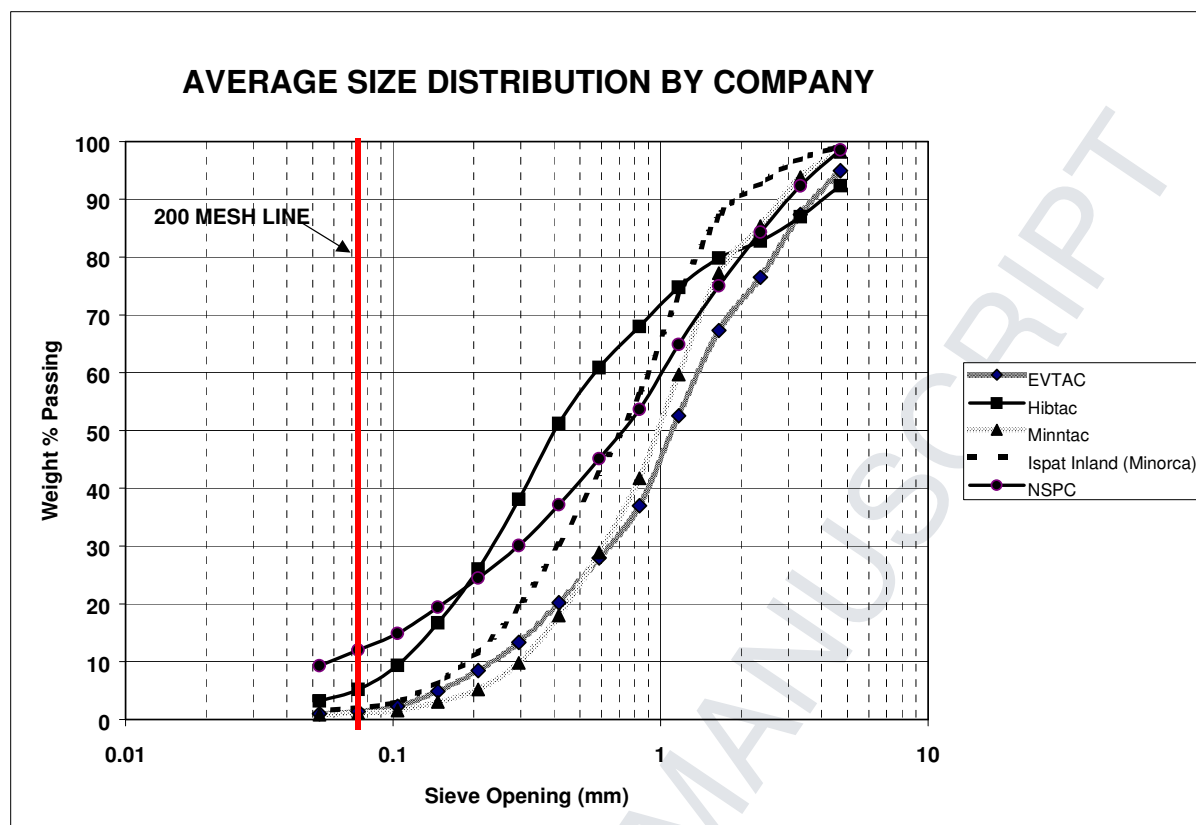
Figure 2: Average coarse tailings size distribution, by company.

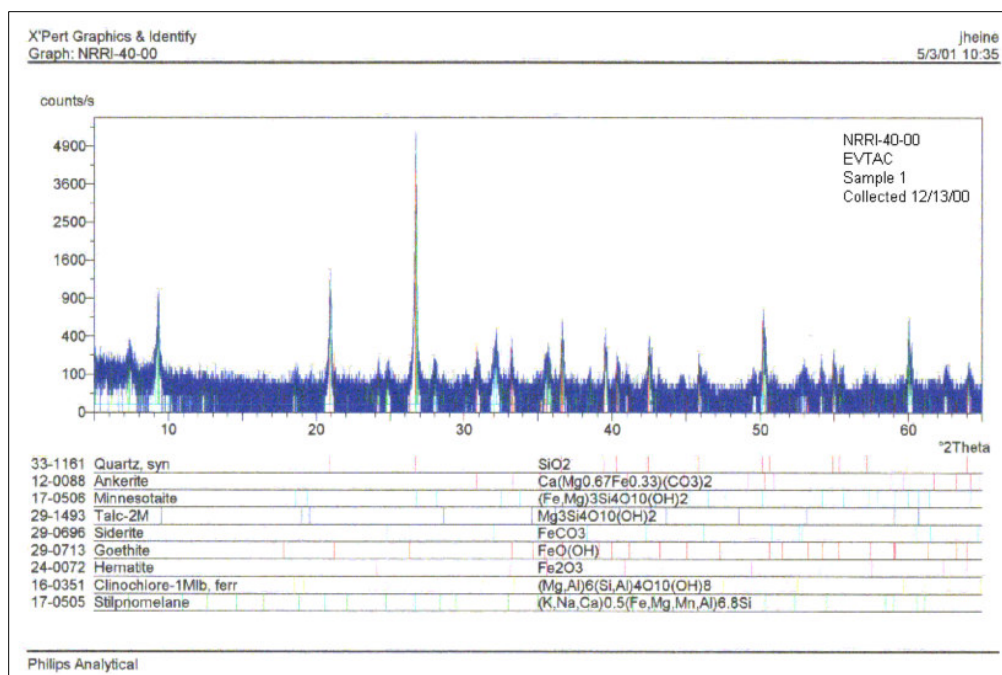
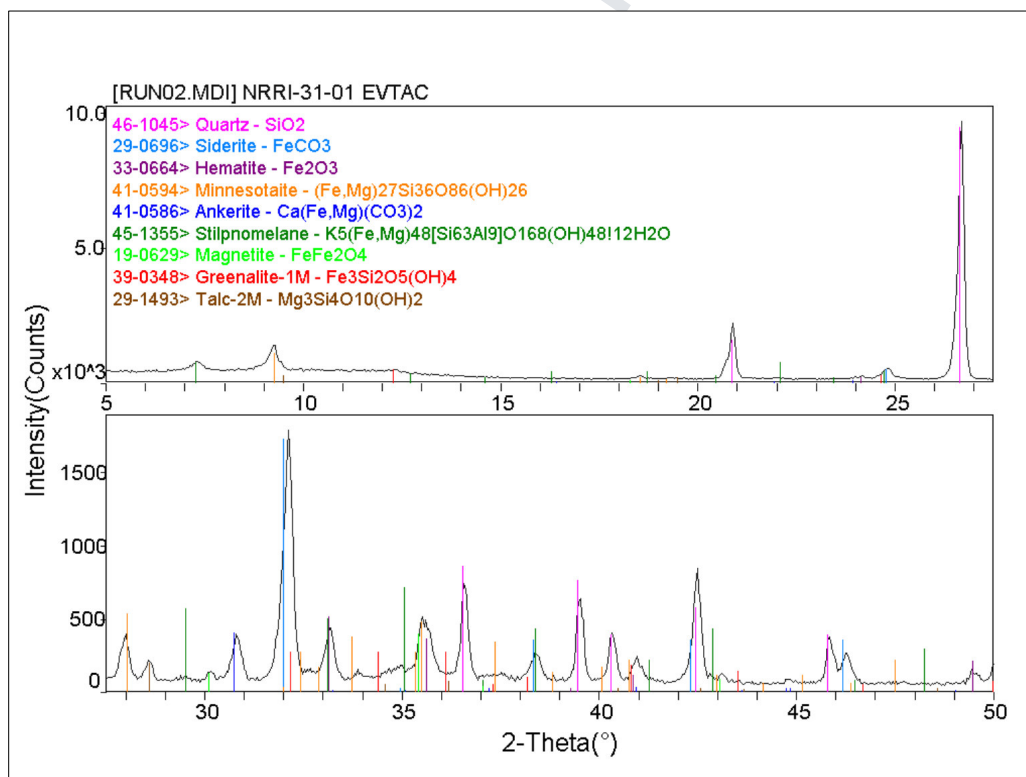
Figure 3a: Typical XRD pattern: NRRI/University of Minnesota, Duluth, analysis.**Figure 3b:** Typical XRD pattern, University of Minnesota Shepherd Laboratories analysis.

Figure 4: Composite quantitative mineralogy of coarse tailings samples, all mines: n=18 samples.

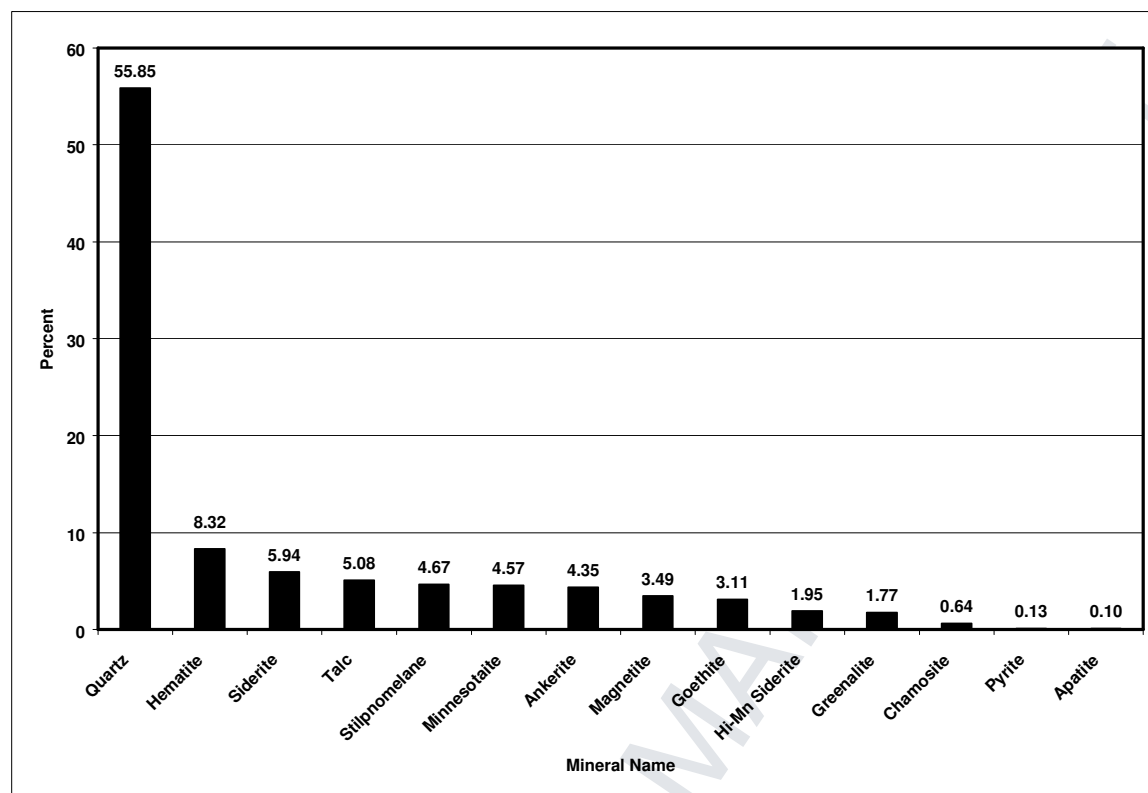


Figure 5: Map of Cliffs Erie (formerly LTV) and Northshore Mining Company properties showing line where grunerite and other amphibole minerals first appear at the eastern end of the BIF (map courtesy of Minnesota Department of Natural Resources, Division of Lands and Minerals, 2003).

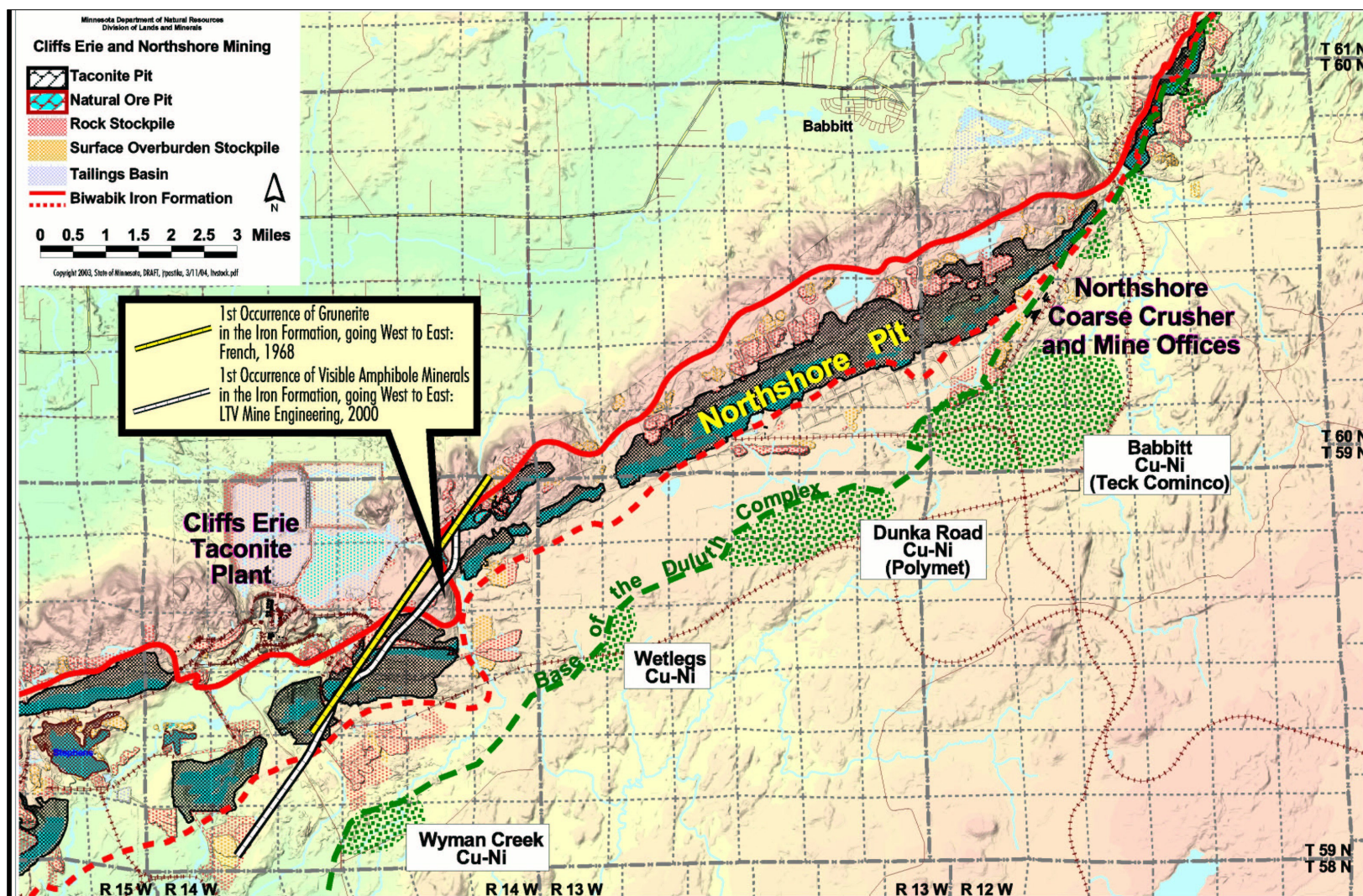


Figure 6a: SEM image of quartz fragment: Ispat Inland (Minorca)

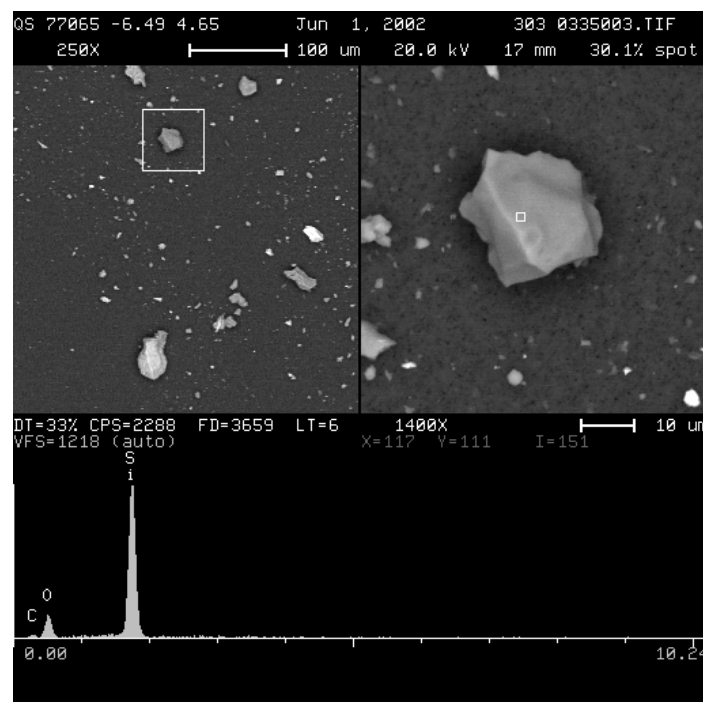


Figure 6b: SEM image of iron-oxide fragment: Ispat Inland (Minorca)

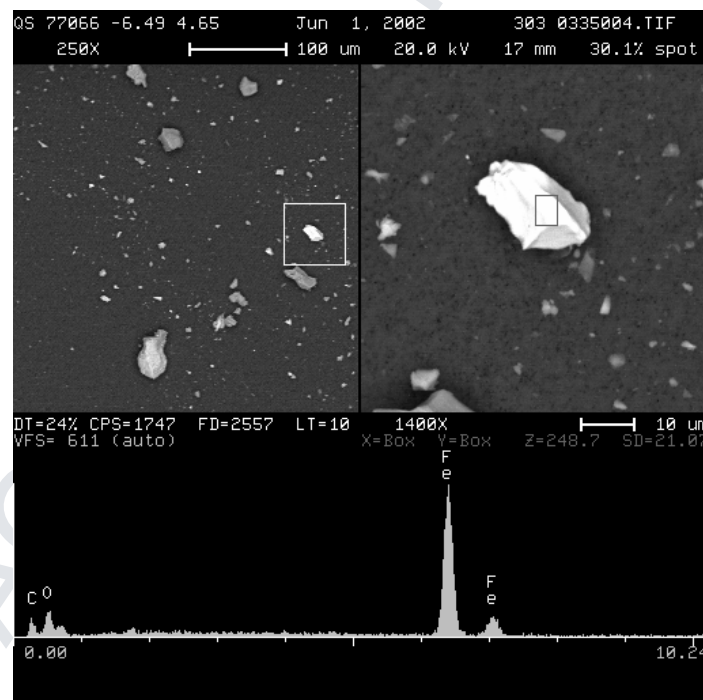


Figure 6c: SEM image of talc fragment: Ispat Inland (Minorca).

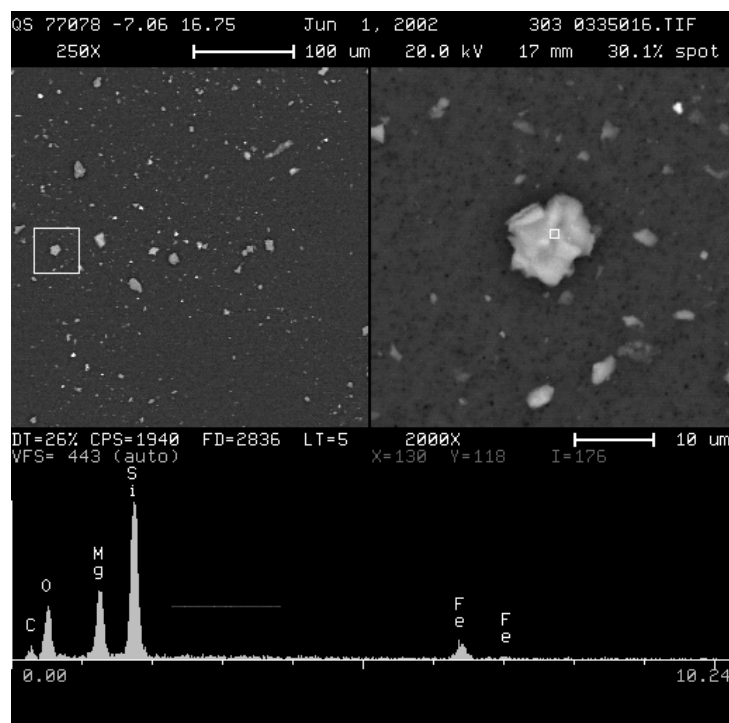


Figure 6d: SEM image of talc cleavage fragment: Ispat Inland (Minorca).

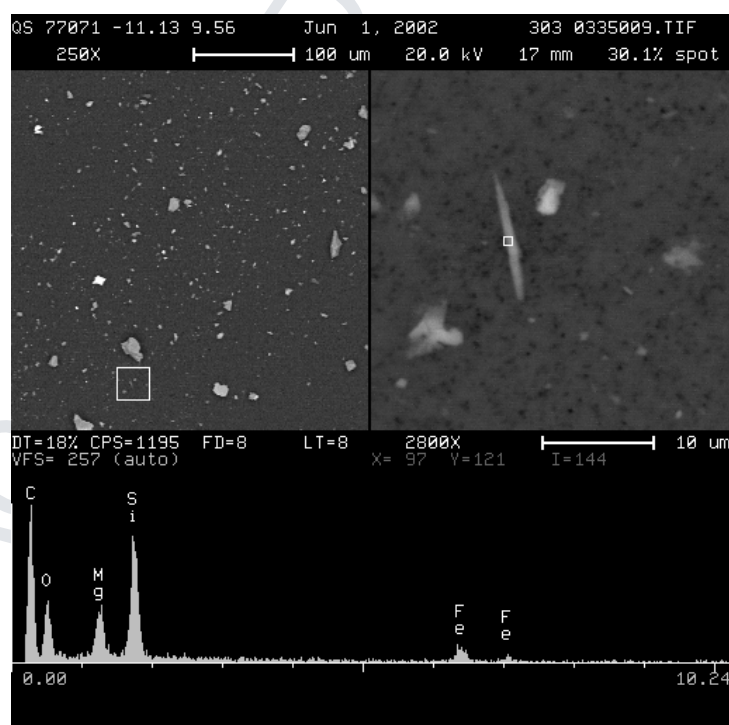


Figure 6e. SEM image of silica-rich (quartz) cleavage fragment: EVTAC

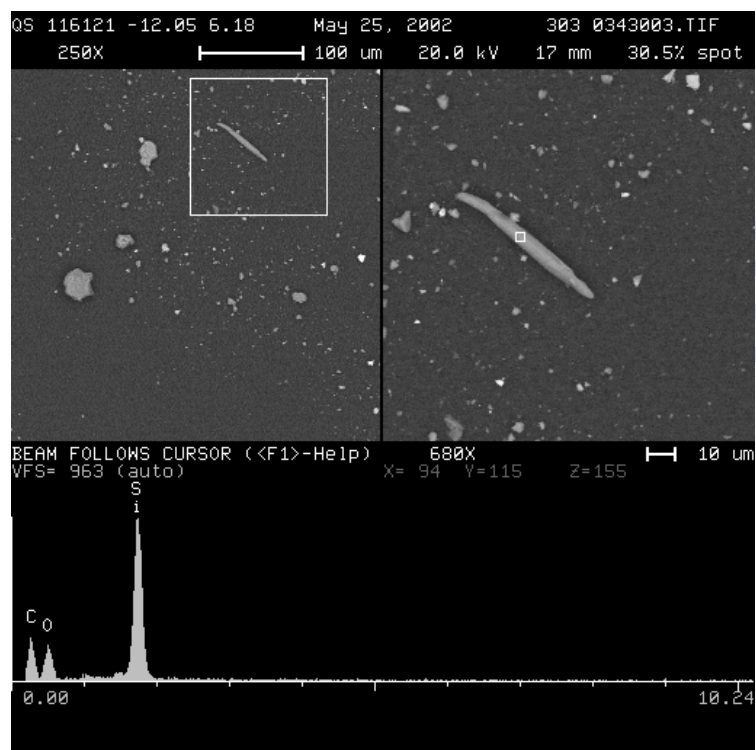


Figure 6f: SEM image of calcium-rich (calcite?) cleavage fragment: EVTAC

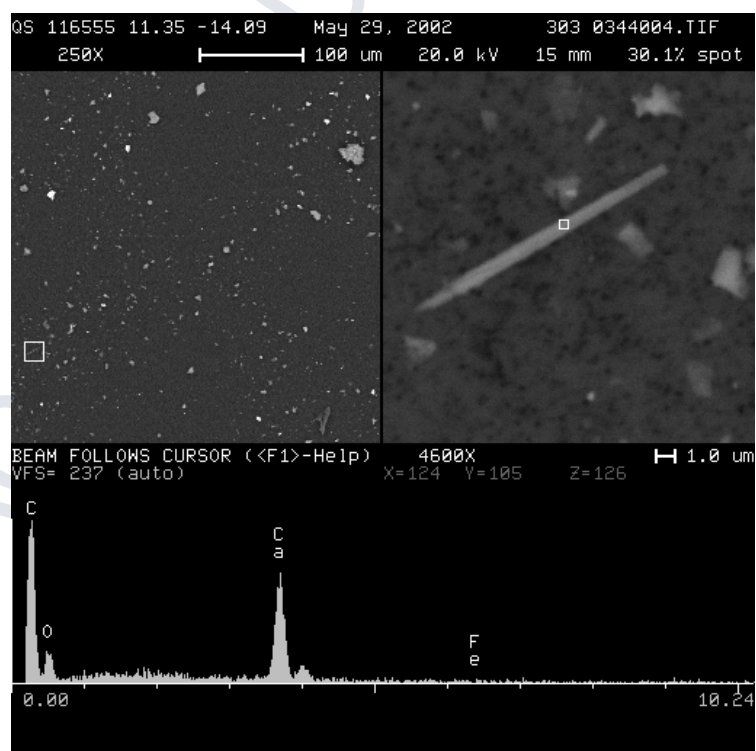


Figure 7a: SEM image of blocky amphibole cleavage fragment.

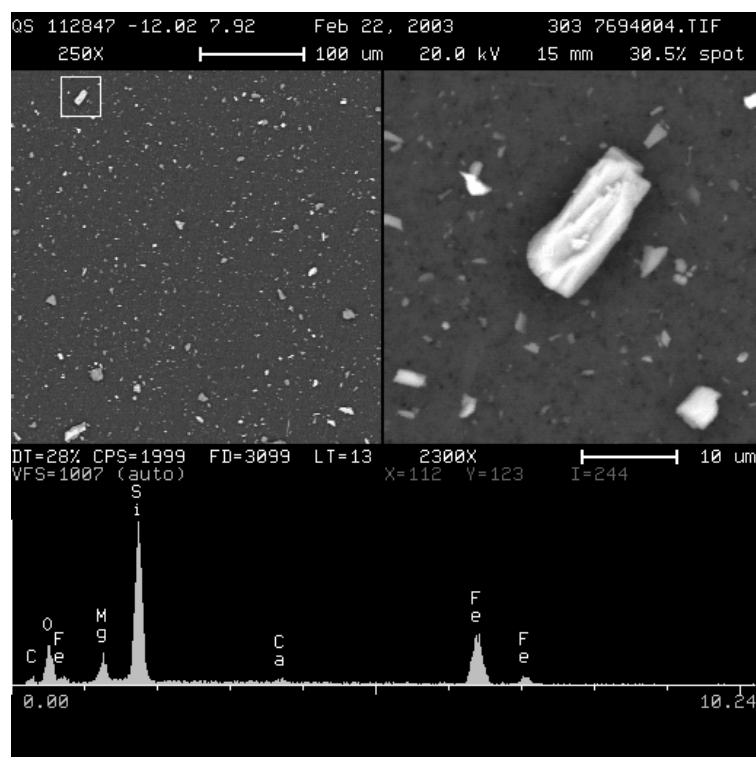


Figure 7b: SEM image of blocky amphibole cleavage fragment.

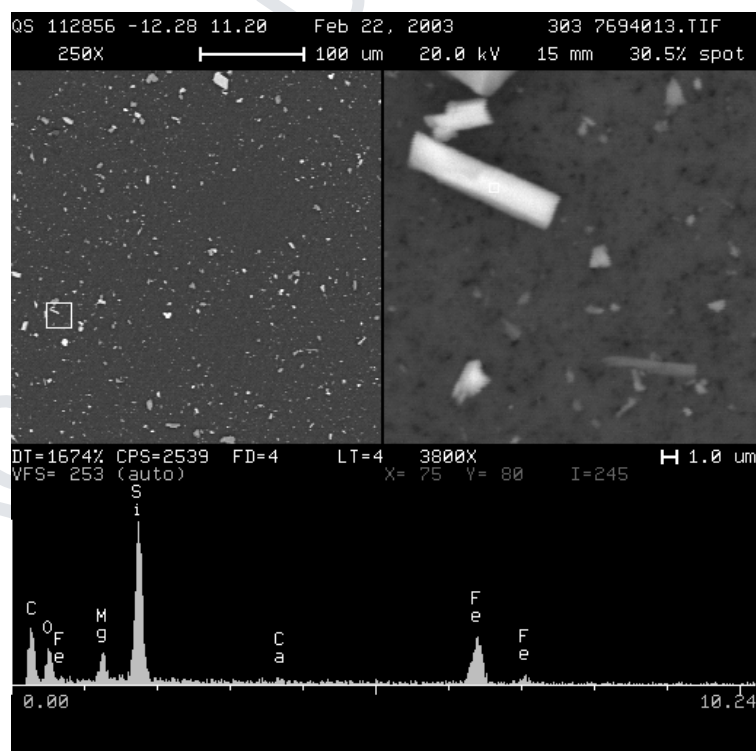


Figure 7c: SEM image of narrow amphibole cleavage fragment.

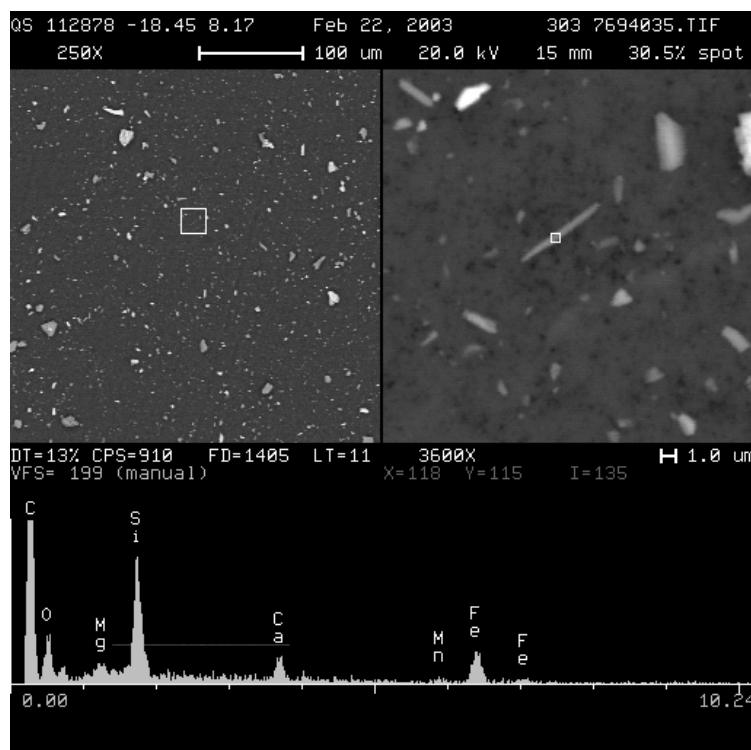


Figure 7d: SEM image of narrow amphibole cleavage fragment.

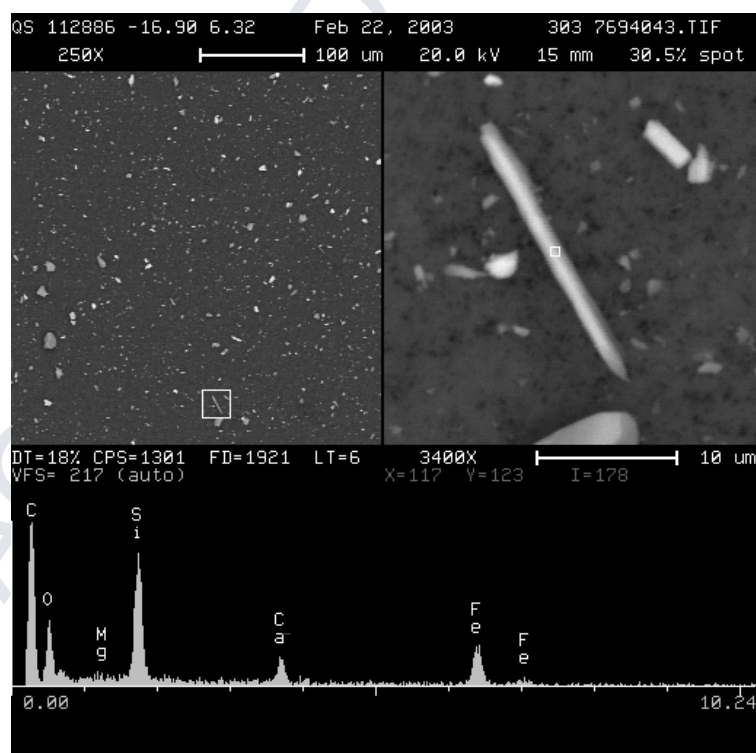


Figure 8: MSHA air sampling Phase Contrast Microscopy (PCM) data: January 10, 2000 to March 25, 2002 (data source: www.msha.gov/asbestos/asbestos.htm).

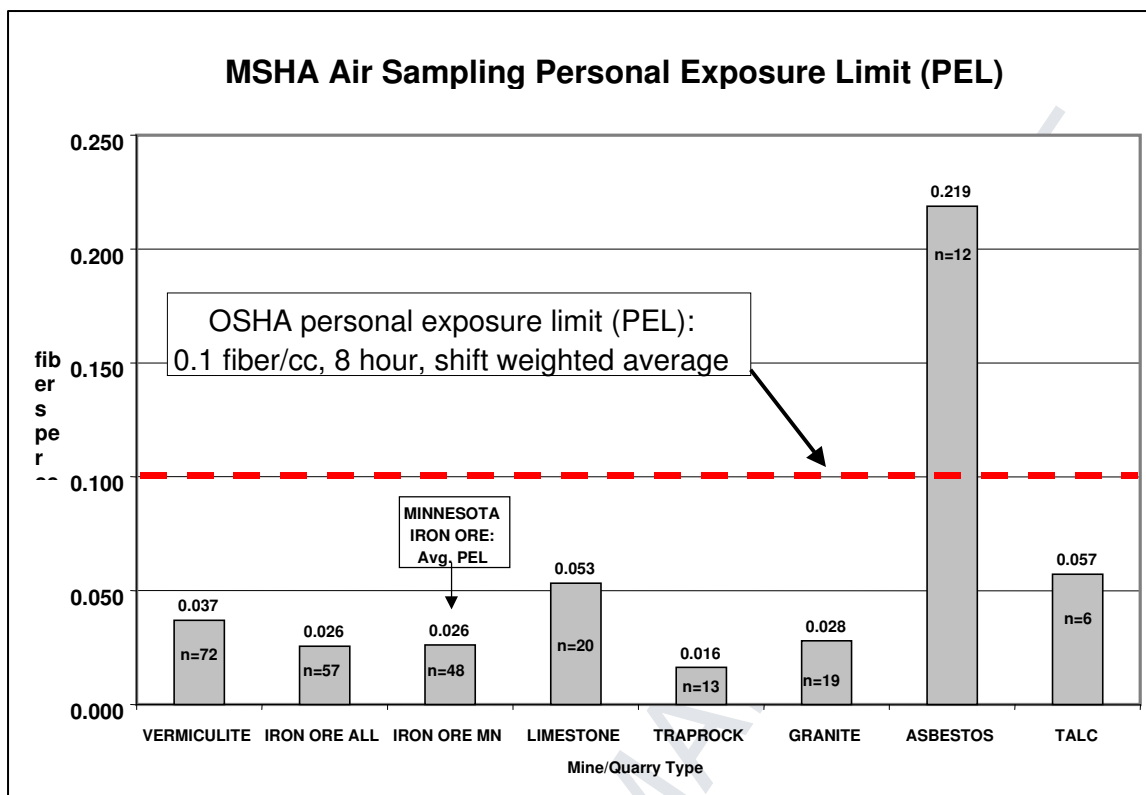


Table 1: Sampling dates and sample numbers.

Event	EVTAC		Hbtac		Mmrtac		Ispat Inland (Mnorca)		NSPC	
	Date	Sample #	Date	Sample #	Date	Sample #	Date	Sample #	Date	Sample #
Sampling 1	12/13/2000	NRRI-40-00	11/16/2000	NRRI-36-00	11/22/2000	NRRI-37-00	12/8/2000	NRRI-39-00		
Sampling 2	3/6/2001	NRRI-10-01	3/9/2001	NRRI-11-01	2/15/2001	NRRI-08-01	3/1/2001	NRRI-09-01		
Sampling 3	6/13/2001	NRRI-31-01	7/24/2001	NRRI-42-01	5/7/2001	NRRI-17-01	6/11/2001	NRRI-30-01	7/9/2001	NRRI-37-01
Sampling 4	9/11/2001	NRRI-49-01	9/12/2001	NRRI-50-01	8/8/2001	NRRI-46-01	9/18/2001	NRRI-52-01	10/1/2001	NRRI-62-01

Table 2: CMRL major oxide chemical analyses used for quantitative mineralogy calculations.

Material	Sample Rec'd. No.	Property	Fe %	Fe++ %	Satmagan MagFe %	TFe ₂ O ₃ %	SiO ₂ %	Al ₂ O ₃ %	CaO %	MgO %	MnO %	Na ₂ O %	K ₂ O %	TiO ₂ %	P ₂ O ₅ %	CO ₂ %	H ₂ O %	Total Oxides %
Coarse Tails	NRRI-40-00	EVTAC	18.4	7.46	2.92	26.31	63.07	0.25	1.25	2.63	0.517	0.017	0.072	0.020	0.044	5.22	1.65	101.05
Coarse Tails	NRRI-10-01	EVTAC	19.4	5.15	5.23	27.74	63.68	0.34	1.06	2.69	0.455	0.017	0.063	0.026	0.041	3.19	1.42	100.72
Coarse Tails	NRRI-31-01	EVTAC	19.7	11.86	1.92	28.17	58.27	0.50	0.97	3.28	0.797	0.059	0.125	0.039	0.060	7.70	1.70	101.66
Coarse Tails	NRRI-49-01	EVTAC	15.3	7.27	1.86	21.87	64.95	0.33	1.66	3.44	0.553	0.080	0.114	0.011	0.034	6.42	1.55	101.02
Coarse Tails	NRRI-36-00	Hibbtac	16.5	8.41	1.79	23.59	63.53	0.37	1.55	3.22	0.637	0.018	0.082	0.011	0.044	6.57	1.57	101.19
Coarse Tails	NRRI-11-01	Hibbtac	17.5	8.28	1.54	25.02	61.79	0.36	1.60	2.78	0.710	0.011	0.073	0.009	0.044	7.10	1.68	101.18
Coarse Tails	NRRI-42-01	Hibbtac	14.6	8.59	1.29	20.87	65.70	0.30	1.63	3.31	0.596	0.053	0.093	0.010	0.039	6.96	1.65	101.22
Coarse Tails	NRRI-50-01	Hibbtac	17.3	7.49	2.84	24.73	63.82	0.22	1.65	2.63	0.634	0.052	0.044	0.019	0.030	5.60	1.60	101.03
Coarse Tails	NRRI-37-00	Minntac	16.4	7.52	2.63	23.45	66.15	0.45	1.12	2.68	0.533	0.025	0.103	0.033	0.041	4.37	1.89	100.84
Coarse Tails	NRRI-08-01	Minntac	15.8	8.67	2.72	22.59	65.46	0.90	1.45	2.77	0.689	0.031	0.126	0.070	0.060	5.24	1.69	101.07
Coarse Tails	NRRI-17-01	Minntac	18.2	5.73	2.52	26.02	65.20	0.33	0.87	2.26	0.617	0.013	0.069	0.020	0.037	3.83	1.42	100.68
Coarse Tails	NRRI-46-01	Minntac	20.3	4.77	2.94	29.02	61.62	0.39	1.61	1.96	0.773	0.019	0.110	0.025	0.046	3.54	1.42	100.53
Coarse Tails	NRRI-39-00	Minorca	16.7	4.40	2.55	23.88	67.28	0.29	1.62	2.04	0.662	0.041	0.078	0.022	0.037	3.45	1.23	100.62
Coarse Tails	NRRI-09-01	Minorca	13.3	4.95	1.80	19.02	70.72	0.27	1.11	3.27	0.646	0.022	0.081	0.013	0.030	4.04	1.48	100.70
Coarse Tails	NRRI-30-01	Minorca	13.5	7.94	3.02	19.30	71.95	0.12	0.97	1.67	0.787	0.052	0.042	0.007	0.025	4.52	1.67	101.12
Coarse Tails	NRRI-52-01	Minorca	17.0	8.89	2.35	24.31	65.53	0.20	1.33	2.57	0.733	0.043	0.036	0.016	0.032	4.77	1.68	101.24
Coarse Tails	NRRI-37-01	NSPC	19.4	5.90	3.10	27.74	61.71	0.30	1.29	3.08	0.656	0.012	0.081	0.016	0.032	4.51	1.40	100.83
Coarse Tails	NRRI-62-01	NSPC	20.6	5.36	2.57	29.45	59.11	0.32	1.59	3.31	0.660	0.014	0.074	0.015	0.032	4.49	1.67	100.74

Table 3: Mineral chemistries used for qualitative mineralogy calculations (see note below).

ASSUMED CHEMICAL COMPOSITIONS OF MINERALS															
CHEMISTRY, percent															
Mineral	Tot. Fe	FeO	Fe ₂ O ₃	SiO ₂	Al ₂ O ₃	CaO	MgO	MnO	Na ₂ O	K ₂ O	CO ₂	Comb. H ₂ O	P ₂ O ₅	S	F
Ankerite	13.99	18.00				29.00	8.50	1.50			43.00				
Apatite, Carb-fluor						52.50		2.50			2.20	0.51	38.00		4.30
Calcite						56.03					43.97				
Chamosite	32.00	36.35	5.37	24.21	19.87		2.44					11.76			
Dolomite						30.41	21.86				47.73				
Fe-Dolomite	9.41	12.11				29.34	12.90	0.77			44.88				
Goethite	62.85		89.86									10.14			
Greenalite	39.48	46.31	5.00	35.59	1.00		2.31					9.79			
Greenalite	39.07	41.00	10.31	35.59	1.00		2.31					9.79			
Greenalite *	38.13	29.00	22.31	33.59	1.00		4.31					9.79			
Hematite	69.94		100.00												
Magnetite	72.36	31.03	68.97												
Minnesotaitite	27.05	34.81	1.45	51.29	0.55		6.26					5.64			
Minnesotaitite	27.05	34.81	2.00	51.29			6.26					5.64			
Pyrite	46.55													53.45	
Quartz				100.00											
Rhodochrosite, (MGS)	1.04	1.34				3.06	0.80	53.08			41.72				
Siderite	40.59	52.24					3.05	4.06			40.65				
Siderite *	36.08	46.44					8.85	4.06			40.65				
Siderite, SIDMN	38.75	48.70					2.60	8.50			40.20				
Siderite, SIDMN*	34.73	44.70					6.60	8.50			40.20				
Siderite, Hi-Mn (MGS)	34.15	43.95					2.72	13.59			39.74				
Siderite, VHi-Mn (MGS)	26.36	33.92					2.45	24.00			39.64				
Stilpnomelane	25.00	27.67	5.00	46.98	5.07		4.22	1.20	0.30	1.74		7.82			
Stilpnomelane	24.66	23.27	9.40	46.98	5.07		4.99	0.59	0.14	1.74		7.82			
Talc				62.35	1.00		31.35					5.30			

Notes: Siderite, SIDMN = manganese bearing siderite; (MGS) = Minnesota Geological Survey determination; and * = mineral form having higher MgO content.